

## GB1477775

Publication Title:

ALKANE SULPHONATE SPRAY PRODUCT

Abstract:

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1477775 Alkali metal alkane sulphonate powders HOECHST AG 27 March 1975 [29 March 1974] 13045/75 Heading C5D A free-flowing spray product in powder form comprises (A) a carrier, which is an inorganic or organic detergent builder, (B) at least one alkali metal alkane sulphonate surfactant, and (C) finely dispersed silica, the content of alkane sulphonate being at least 15% by wt. referred to the combined wt. of (A) and (B) and the content of SiO<sub>2</sub> being from 0.2 to 6% by wt. based on (B). (A) may be sodium carbonate, sodium metasilicate, sodium tripolyphosphate, sodium sulphate, sodium citrate, potassium carbonate, potassium polyphosphate, potassium sulphate, ammonium sulphate or ammonium polyphosphate. (B) preferably contains C<sub>12</sub> to C<sub>18</sub> carbon atoms and is present in quantities of at least 20% (based on (A) plus (B)). The powder is produced by spray-drying at a temperature of 100 C. to 350 C. an aqueous slurry of (A), (B) and (C) above. Preferably, the silica is in the form of a fine dispersion having a bulk wt. of 60 to 400 g. per litre. The powder may be incorporated into detergent compositions containing conventional additives such as soaps, bleach and optical brighteners. Data supplied from the esp@cenet database - Worldwide

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## (54) ALKANESULPHONATE SPRAY PRODUCT

(71) We, HOECHST AKTIENGESellschaft, a Body Corporate organised under the laws of the Federal Republic of Germany of 6230 Frankfurt/Main 80 Germany, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a spray product containing an alkali metal alkanesulphonate as surfactant and to a process for its preparation.

It has been proposed to prepare detergent powders by spray drying mixtures of active detergent substances, e.g. soaps, alkarylsulphonates, alkenesulphonates and alkanesulphonates, together with other predominantly inorganic additives as carriers. Whereas spray drying of mixtures of the first three above-mentioned detergent substances and inorganic additives generally yields free-flowing, pourable powders which do not cake together on storing, the products of spray drying mixtures of alkane-sulphonates and inorganic additives generally have poor storage properties if the detergent content is high. Since alkanesulphonates are hygroscopic, spray products produced therefrom with a high content of alkanesulphonates tend to cake or even become sticky as a result of water absorption from the atmosphere or from the inorganic additives which contain water of crystallisation. For this reason alkanesulphonates have hitherto been used satisfactorily only in solution e.g. in liquid cleaning materials and bath additives.

There have been numerous attempts to overcome the problem of the poor storage properties of alkanesulphonates and to facilitate the preparation of spray products containing alkanesulphonates by adding small quantities of so-called "anti-caking agents" to the spray mixture. Thus benzene-

sulphonates, toluenesulphonates and magnesium silicate have for example been proposed as "anti-caking agents". In addition, high-melting, finely divided powders which are not hygroscopic have been added to the spray-dried products. Such powders act purely mechanically by coating the individual granules of the spray product, thus preventing them from sticking to one another. It is also possible to reduce caking on storage by greatly increasing the proportion of carrier to detergent but this is commercially undesirable in that the detergent strength of the powder is reduced.

According to the present invention we now present a free-flowing spray-dried powder comprising A) a carrier comprising at least one salt of an inorganic or organic acid and an inorganic base, B) at least one alkali metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate being at least 15%, preferably at least 20% by weight referred to the combined weight of carrier and alkanesulphonate and the content of silica being from 0.2 to 6%, preferably from 0.3 to 4% by weight referred to the weight of alkanesulphonate.

The spray-dried powders according to the invention are in general free-flowing, non-hygroscopic and thus non-caking. They may be prepared by spray-drying an aqueous composition containing A) a carrier comprising at least one salt of an inorganic or organic acid and an inorganic base, B) at least one alkali metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate in the composition being at least 15%, preferably at least 20% by weight referred to the combined weight of the carrier and alkanesulphonate and the content of silica being from 0.2 to 6%, preferably from 0.3 to 4% by weight referred to the weight of alkanesulphonate at a temperature from 100 to 350°C, preferably from 120 to 320°C,

which process constitutes a further feature of the invention.

The alkanesulphonate component of the spray-dried powders according to the invention preferably comprises alkanesulphonates containing from 12 to 18 carbon atoms in the alkyl group and the alkali metal is preferably sodium or potassium.

The silica is conveniently added to the aqueous spray composition in the form of a dispersion having a bulk weight of from 60 to 400g per litre.

The carrier generally should have a melting point of at least 150°C. Salts of non-corrosive acids are preferred, particularly their sodium salts such as sodium carbonate, sodium metasilicate, sodium tripolyphosphate, sodium sulphate and sodium citrate. Small quantities of sodium chloride may also be contained in the spray mixture, for example as impurity introduced into the above-named sodium salts during their preparation. Other suitable carrier salts include for example potassium and ammonium sulphate, potassium carbonate and a potassium and an ammonium polyphosphate. Mixtures of carrier salts may also be used. The carrier is preferably soluble in water at 100°C in an amount of at least 30% by weight.

The spray-dried powder may if desired contain other additives conventionally used in washing powders, for example optical brighteners, bleaches and soaps.

The process of preparing the spray-dried powder according to the invention may be effected in a conventional spray plant. Thus for example the hot aqueous spray composition may be atomised by passage through a nozzle and brought into contact with a flow or counterflow of hot gases. The aqueous composition is preferably as concentrated as possible for the spray-drying method. Water contained in the spray composition evaporates and a spray product is obtained which consists of approximately spherical, non-caking particles. The bulk weight and water content of the spray product may be adjusted by selecting appropriate spray conditions, the spray temperature being particularly important.

The spray-dried powders according to the invention may be used as components of surfactant compositions optionally in admixture with the aforementioned additional additives. They may also be used as lubricants or carriers in cosmetic preparations. It is also possible to use the process according to the invention for a one-step preparation of commercial detergent compositions.

The following Examples serve to illustrate the preparation of spray-dried powders according to the invention.

#### Example 1

A 35% aqueous spray solution containing a mixture of sodium sulphate and sodium alkanesulphonates (containing, on average, 15 carbon atoms in the alkyl group) in a weight ratio of 1.5:1 is prepared. 4% by weight (based on the weight of alkanesulphonates) of a fine dispersion of silica with a bulk weight of 100g per litre is dispersed in the aqueous spray solution. The mixture is then sprayed at a temperature between 315 and 340°C. A white, free-flowing, non-hygroscopic spray-dried powder with a water content of about 7% by weight and a bulk weight of 300 to 400g per litre and which does not cake on storage is obtained.

#### Example 2

A 32% aqueous spray solution containing a mixture of sodium alkanesulphonates containing 12 to 18 carbon atoms in the alkyl groups and sodium tripolyphosphate in a weight ratio of alkylsulphonates to tripolyphosphate of 1:3 is prepared. 0.375% by weight (based on the weight of alkanesulphonates) of a fine silica dispersion with a bulk weight of 100g per litre is dispersed therein. The mixture is sprayed as in Example 1 to yield a white, free-flowing, non-hygroscopic spray-dried powder with a bulk weight of 300 to 400g per litre and a water content of 10% by weight and which does not cake on storage.

#### Example 3

A 45% aqueous spray solution containing a mixture of sodium alkanesulphonates containing predominantly alkanesulphonates with 12 to 15 carbon atoms, sodium sulphate and sodium tripolyphosphate in a weight ratio of alkanesulphonates to sulphate to tripolyphosphate of 1:2:2 is prepared. 1.1% by weight (based on the weight of alkanesulphonates) of a fine silica dispersion with a bulk weight of 200g per litre is dispersed therein. The mixture is sprayed as in Example 1 to yield a white, free-flowing, non-hygroscopic spray-dried powder with a bulk weight of 180 to 250g per litre and a water content of 7% by weight and which does not cake on storage.

#### Example 4

A spray-dried powder is prepared analogously to Example 1 except that the aqueous spray mixture is sprayed at a temperature of 290 to 300°C. A white, free-flowing, non-hygroscopic spray-dried powder with a bulk weight of 300 to 400g per litre and a water content of about 8% by weight and which does not cake on storage is obtained.

#### Comparison Example A

Spray products are prepared analogously to Examples 1 to 2 but omitting the silica dispersion. The products are white and initially free-flowing but cake on storage.

#### Comparison Example B

A 33% aqueous spray solution containing

a mixture of sodium alkanesulphonates containing 12 to 18 carbon atoms in the alkyl groups and sodium tripolyphosphate in a weight ratio of alkanesulphonates to tri-  
5 polyphosphate of 2:15 and containing no silica dispersion is sprayed analogously to Example 1 to yield a white, free-flowing product which does not cake on storage and which has a bulk weight of 300 to 400g per  
10 litre and a water content of 15% by weight.

*Comparison Example C*

A spray-dried powder is prepared analogously to comparison Example B except that tetrasodium pyrophosphate is used in  
15 the place of sodium tripolyphosphate. A white, free-flowing product which does not cake on storage and which has a bulk weight of 300 to 400g per litre and a water content of 15% by weight is obtained.

20 WHAT WE CLAIM IS:—

1. A free-flowing spray-dried powder comprising A) a carrier comprising at least one salt of an inorganic or organic acid and an inorganic base, B) at least one alkali  
25 metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate being at least 15% by weight referred to the combined weight of carrier and alkanesulphonate and the content of silica being from 0.2 to 6% by  
30 weight referred to the weight of alkanesulphonate.

2. A spray-dried powder as claimed in claim 1 wherein the content of silica is from  
35 is from 0.3 to 4% by weight referred to the weight of alkanesulphonate.

3. A spray-dried powder as claimed in claim 1 or claim 2 wherein the carrier has a melting point of at least 150°C.

4. A spray-dried powder as claimed in any of the preceding claims wherein the carrier is sodium carbonate, sodium metasilicate, sodium tripolyphosphate, sodium sulphate, sodium citrate, potassium carbonate,  
45 a potassium polyphosphate, potassium sulphate, ammonium sulphate or an ammonium polyphosphate.

5. A spray-dried powder as claimed in claim 3 or claim 4 wherein the carrier is soluble in water at 100°C in an amount of  
50 at least 30% by weight.

6. A spray-dried powder as claimed in any of the preceding claims wherein the alkanesulphonate component comprises an alkanesulphonate containing from 12 to 18  
55 carbon atoms.

7. A spray-dried powder as claimed in any of the preceding claims wherein the alkanesulphonate component comprises a sodium or potassium alkanesulphonate.  
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8. A spray-dried powder as claimed in any of the preceding claims wherein the content of alkanesulphonate is at least 20% by weight referred to the combined weight  
65 of carrier and alkanesulphonate.

9. A spray-dried powder as claimed in any of the preceding claims which additionally contains at least one further ingredient selected from optical brightening agent,  
70 bleaches and soaps.

10. A spray-dried powder as defined in claim 1 substantially as herein disclosed.

11. A spray-dried powder as defined in claim 1 substantially as herein disclosed.

with particular reference to Example 1 to 3.  
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12. A spray-dried powder as defined in claim 1 substantially as herein described with particular reference to Example 1 to 3.

13. A process for the preparation of a free-flowing spray-dried powder containing  
80 an alkali metal alkanesulphonate as surfactant which comprises spray-drying at a temperature of from 100 to 350°C an aqueous composition containing A) a carrier comprising at least one salt of an in-  
85 organic or organic acid and an inorganic base, B) at least one alkali metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate in the composition being at least 15%  
90 by weight referred to the combined weight of the carrier and alkanesulphonate and the content of silica being from 0.2 to 6% by weight referred to the weight of alkanesulphonate.

14. A process as claimed in claim 13 wherein the composition is spray dried at a temperature of from 120 to 320°C.

15. A process as claimed in claim 13 or claim 14 wherein the content of silica in  
100 the composition is from 0.3 to 4% by weight.

16. A process as claimed in any of claims 13 to 15 wherein the silica is introduced into the aqueous composition in the  
105 form of a fine dispersion having a bulk weight of 60 to 400g per litre.

17. A process for the preparation of a spray-dried powder according to claim 13 substantially as herein described.  
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18. A process for the preparation of a spray-dried powder according to claim 13 substantially as herein described with reference to Examples 1 to 4.

19. A process for the preparation of  
115 spray-dried powder according to claim 13 substantially as herein described with reference to Examples 1 to 3.

20. A free-flowing spray-dried powder whenever prepared by a process as claimed  
120 in any of claims 13 to 18.

21. A surfactant composition comprising a free-flowing spray-dried powder as claimed in any of claims 1 to 12 and 20.

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